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# PVT Properties of Polymers for Injection Molding

Jian Wang

*School of Chemical Engineering and Environment, Beijing Institute of Technology,  
China*

## 1. Introduction

PVT (Pressure-Volume-Temperature) properties of polymers are important for both engineering and polymer physics. Fig.1 shows the typical PVT diagrams of an amorphous (a) and semi-crystalline polymer (b). PVT diagram describes the specific volume as a function of pressure and temperature. Specific volume increases with the temperature increasing. There is a thermal transition in the polymer. The primary amorphous transition of any polymer is known as its glass transition temperature,  $T_g$ . While  $T_g$  is not a sharp transition, the data from below and above  $T_g$  will show an intersection that is generally accepted as being  $T_g$ . As shown in Fig. 1, semi-crystalline polymer exhibits a different thermal response than amorphous polymer. For the amorphous polymer,  $T_g$  is clearly seen as the temperature where the polymer goes from a solid to a melt. The rate of expansion per temperature increment is much smaller in the solid state than in the melt state. By contrast, the semi-crystalline polymer contains sufficient crystallinity to maintain structural continuity above  $T_g$ . While the amorphous content in this polymer exhibits a  $T_g$ , the crystal structure allows characterization up to nearly the temperature where the crystals melt.

Polymer PVT data become increasingly important in their value in material science. The excess usage of PVT data can be summarized in at least eight major areas (Berry et al., 1998; Hess, 2004):

- Prediction of polymer-polymer miscibility;
- Prediction of service performance and service life of polymeric materials and components on the basis of free volume concepts;
- Correlation of the reducing parameters of equations of state (EOS) with molecular structures;
- Evaluation of start and progress of chemical reactions in polymer melts in the cases when volume effects accompany the reaction;
- Materials properties of systems in contact with solvents or gases;
- Investigation of the nature of phase transitions;
- Optimizing of processing parameters instead of establishing such parameters by trial and error;
- Calculation of the surface tension of polymer melts.

Injection molding is the most common technique for the mass production of complex shaped products that require accurate dimensions. In injection molding process, some

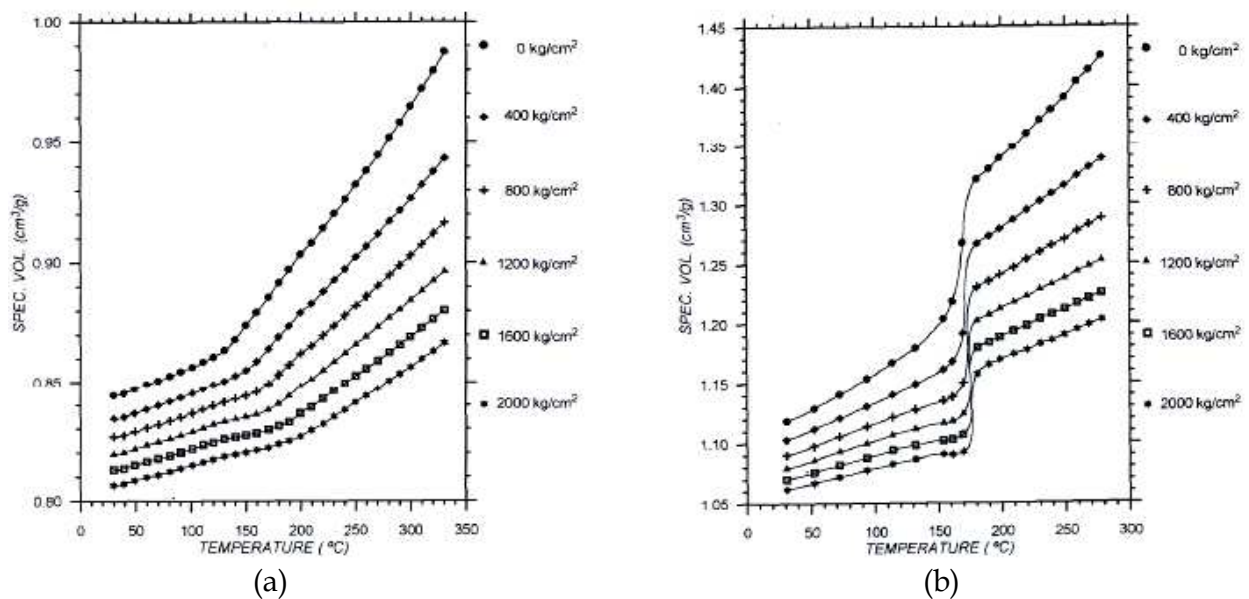


Fig. 1. The typical PVT diagrams of an amorphous (a) and semi-crystalline polymer (b) (Zoller & Walsh, 1995).

defects such as shrinkage, warpage and sink marks will cause thermally induced stresses, and affect both dimensional accuracy and long-term dimensional stability. Software such as Moldflow (Autodesk, Inc., San Rafael, CA) or Moldex 3d (CoreTech System Co., Ltd., Taipei, China) seeks to reduce the risk of producing parts with shrinkage or warpage in plastic components by providing quantitative predictions based on reliable data. In numerical simulation of the injection molding process, one of the important input data set to these software packages is PVT data (Brown & Hobbs, 1998). On the other hand, process analyses have revealed that optimum processing conditions can be achieved by application of PVT diagrams (see Fig.1), particularly if PVT diagrams are used in conjunction with computer control of the process (Menges & Thienel, 1977). Therefore, it is necessary to get more accurate PVT data for more accurate prediction, evaluation, optimization and calculation.

This chapter is an introduction to PVT properties of polymers for injection molding. It is divided into three sections: different measurements of polymer PVT properties are introduced in detail, followed by the testing modes, and then two main application areas of polymer PVT data are illustrated in detail, including numerical simulation and process control.

## 2. Different measurements of polymer PVT properties

In this section, different measurements of polymer PVT properties are introduced in detail, including conventional measurements (piston-die technique and confining-fluid technique), some improved experimental techniques considering the effect of cooling rate, shear rate and pressure, on-line techniques using injection molding machine or extruder, etc.

### 2.1 Conventional measurements

Using a dilatometer is the most common technique to measure the bulk specific volume as a function of temperature and pressure of polymers. There are two principally different

conventional techniques performing PVT measurements: the piston-die technique and the confining-fluid technique.

2.1.1 Piston-die technique

The piston-die technique (Fig.2a): The material is enclosed and pressurized in a rigid die using a piston which is tightly fitted into the die. During the measuring cycle the volume of the material is recorded by measuring the displacement of the piston. Both temperature and pressure can be varied. The advantage of this technique is the simplicity of the design that can be achieved. The disadvantage is that the pressure applied is not hydrostatic because the material sticks to the wall (He & Zoller,1994). Other problems are the possible leakage between the piston and the die and the formation of voids in the sample when solidifying. The piston-die technique was applied by Chang et al. (1996) who used a PVT-100 apparatus from SWO Germany (see Fig. 3).

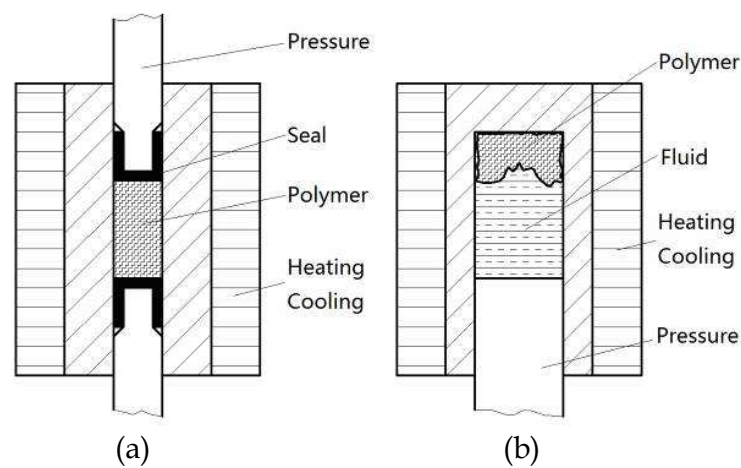


Fig. 2. Sketch diagrams of piston-cylinder technique (a) and confining-fluid technique (b).

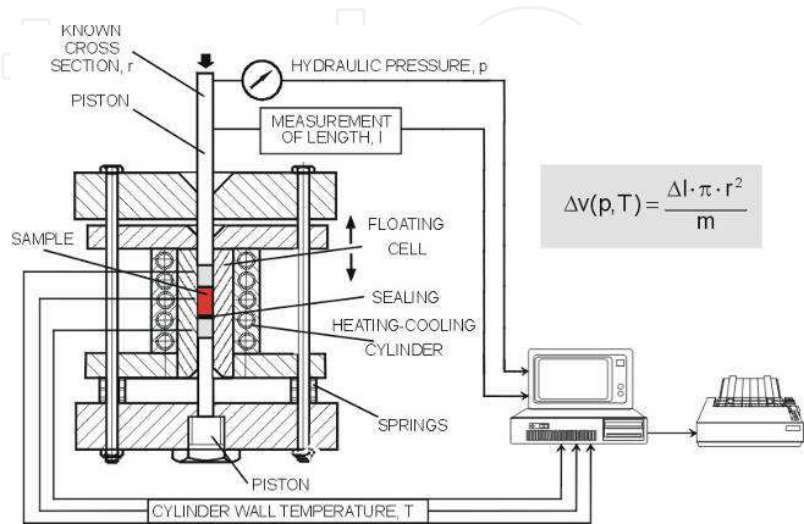


Fig. 3. Principle schematic diagram of PVT100 (SWO Polymertechnik GmbH, 1998).

### 2.1.2 Confining-fluid technique

The confining-fluid technique (Fig.2b): The sample is enclosed in a rigid sample chamber, and it is submerged into a fluid (mercury or silicon oil). The cell is closed using a flexible wall or bellows. The bellows is used to apply hydrostatic pressure to the fluid and polymer by reducing the sample chamber volume, and sensing the cumulative volume change of fluid and polymer. The absolute specific volume of the polymer can be obtained by correcting the relative volume difference with the specific volume of the confining fluid. Both pressure and temperature can be varied. The advantages of this technique are:

- Pressure is purely hydrostatic as the sample is surrounded by the confining fluid in both a melted and solid state;
- There is an absence of leakage and friction.

The disadvantages are:

- The volumetric changes measured are not that of the polymeric sample only;
- Sealing of the pressurized fluid and reactions may occur between polymers and the confining fluid.

Quach & Simha (1971) constructed an apparatus based on the confining-fluid technique. Mercury was employed as the confining liquid. The operating range is  $0 \leq T \leq 200^\circ\text{C}$  and  $1 \leq P \leq 200\text{MPa}$ . Calibration with benzene and mercury showed an accuracy of  $\pm 2 \times 10^{-4} \text{ cm}^3/\text{g}$  in the measurement of the specific volume change. Zoller et al. (1976) developed an apparatus which was based on the classical confining-fluid technique. The apparatus (Gnomix, Inc., Boulder, CO) shown in Fig. 4 was used by Moldflow to get PVT data. The PVT instrument has some advantages compared to a capillary rheometer if accurate numbers are desired ( $0.0001 \text{ cm}^3/\text{g}$ ) (Zoller & Fakhreddine, 1994).

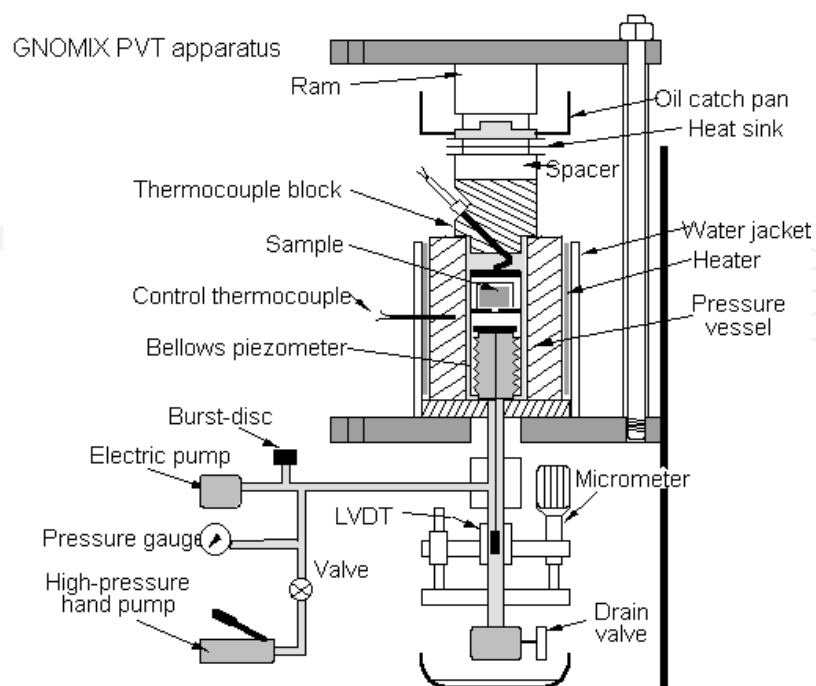


Fig. 4. Gnomix PVT testing device developed by Zoller et al. (1976).



Barlow (1978) developed a bellows dilatometer at pressures up to 280 MPa but temperatures only up to 55 °C. The experimental system used a bellows-type dilatometer for measuring sample volume changes. As shown in Fig.5, the below cell was designed expressly for the purpose of measuring volume changes of solid polymers surrounded by mercury.

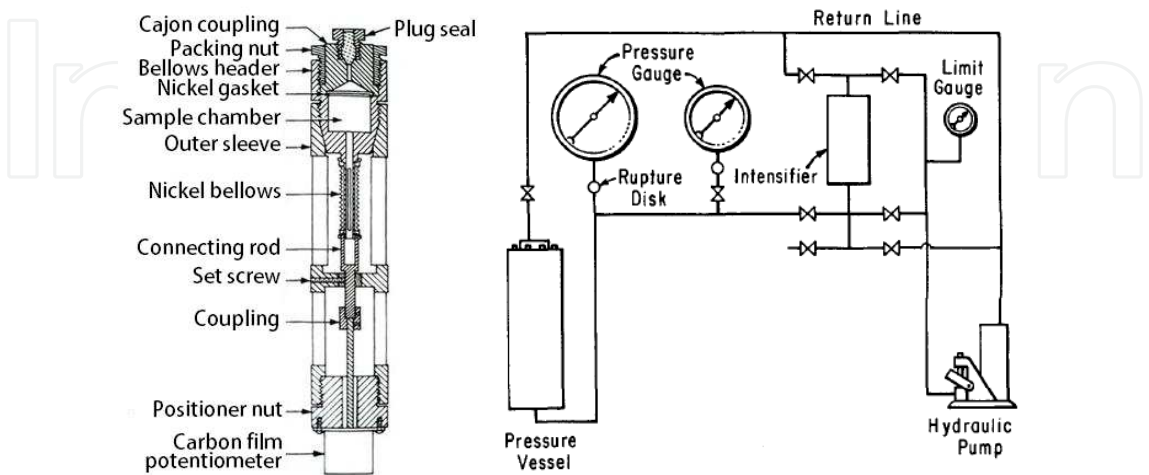


Fig. 5. PVT testing setup developed by Barlow (1978).

Sato et al. (1997) developed an apparatus (Fig. 6) using a metal bellows at temperatures from 40 to 350 °C and pressures up to 200 MPa. The experimental uncertainty of specific volumes was estimated to be within  $\pm 0.2\%$ . The effects of a sample cup and sample forms were investigated. The use of the sample cup showed a little effect on the measurments of PVT properties for both samples. The shape (pellet and pillar) of the samples caused a small difference in the specific volumes only under high temperatures and low pressures.

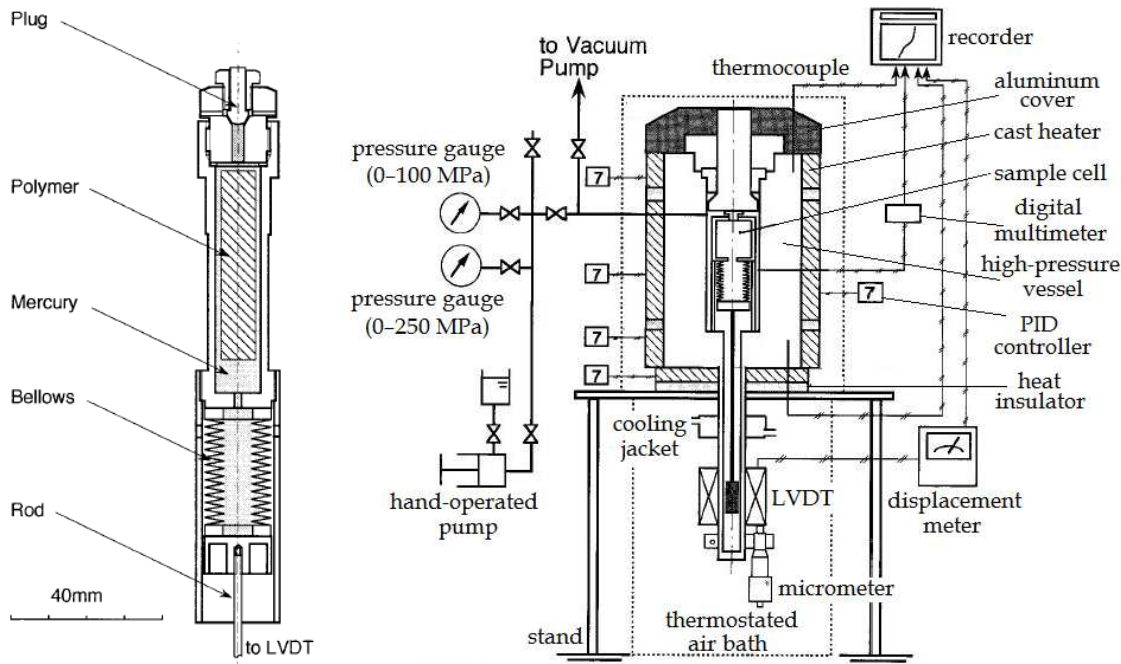


Fig. 6. Schematic diagram of the PVT testing setup developed by Sato et al. (1997).

It has been shown that differences between the piston-die and confining-fluid technique in methods or measurement principles do not lead to differences in measured values bigger than 4% (Sato et al., 1997).

## 2.2 Improved measurements

The material properties of polymers show a distinct time dependence, i.e., depending on the rate of change of state, they show different thermal material behaviour, particularly in the transition range between melt and solid. Thus, the values of specific volume differ for low and high cooling rates (Fig. 7) (Menges & Thienel, 1977).

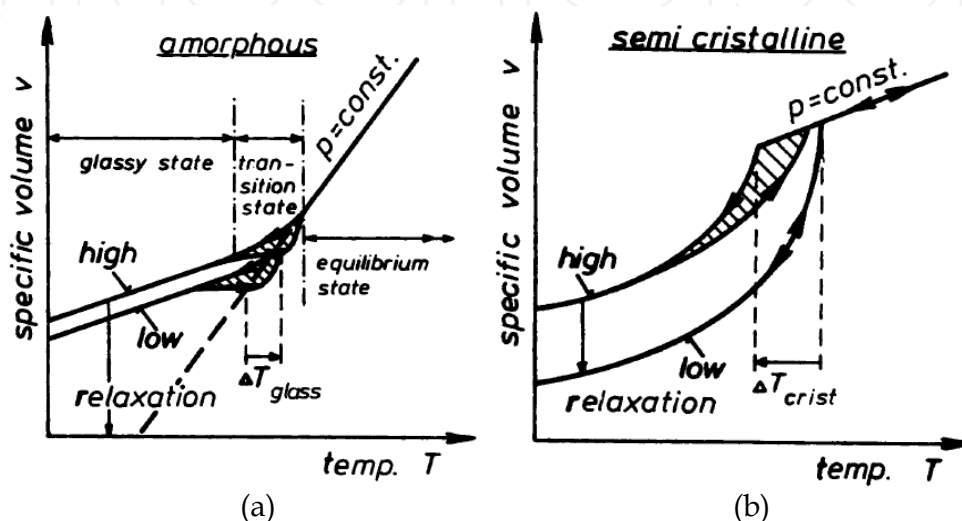


Fig. 7. PVT diagrams of an amorphous (a) and semi-crystalline polymer (b) under different cooling rates (Menges & Thienel, 1977).

As shown in Fig. 7, amorphous polymer reaches its equilibrium degree of order above the glass temperature  $T_g$  within the time of measurement. This is not possible at temperatures below  $T_g$ ; the melt will then pass over from a state of equilibrium to one of disequilibrium, i.e., to the frozen-in or glassy state. This state is distinguished, in general, by a greater heat content (enthalpy). In this range, there is a smaller coefficient of thermal expansion compared to the molten state. In the glassy state, the molecules retain the state of order they had reached at the glass temperature. At higher cooling rates the glass temperature is shifted to higher values. Since at higher glass temperatures, a lower state of order prevails, a high cooling rate causes a lower density or a greater specific volume than do lower cooling rates.

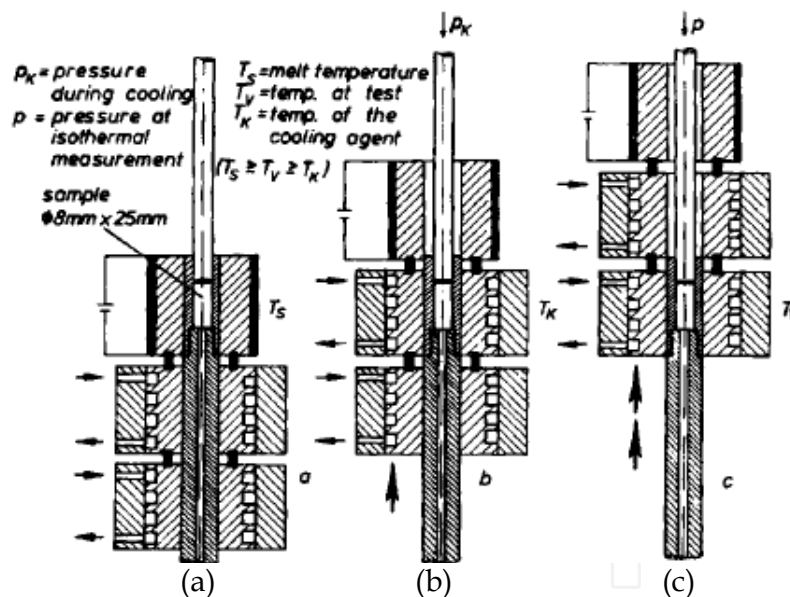
With semi-crystalline polymer, crystallization takes place below the crystallization temperature (beginning of solidification). If the time available for crystal growing decreases due to an increased cooling rate, crystallization will be slowed down or reduced. This becomes evident in a shift of the crystallization temperature to lower values (melt begins solidifying at lower temperatures) and in a growing reduction in density (increase of specific volume) below this temperature.

Apart from the effects of cooling rates during the transition between melt and solid, specific volume below the glass temperature or crystallization temperature is furthermore influenced by:

- The pressure during cooling.
- The testing modes of operation: isobaric measurement and isothermal measurement.

If specific volume data are to be used to gain better understanding of what is happening during industrial processing of polymers or as input for constitutive modelling used in simulation software, it is necessary to measure specific volume at conditions comparable to industrial processing conditions. Conventional techniques can only be used at relatively low cooling rates. However, plastic processing, such as injection molding, is a rapid, high pressure process where both cooling rate and pressure play critical roles in the final component dimensions. Since high cooling rates are present during injection molding, some improved PVT testing techniques have been designed.

Menges and Thienel (1977) developed a new measuring instrument (see Fig. 8) which made it possible to determine such PVT diagrams using an adjustable barrel chamber under normal processing conditions. Piccarolo (1992) used a different method to measure the specific volume of semicrystalline polymers at high cooling rates above 1200 °C/min. Bhatt and McCarthy (1994) developed a PVT apparatus for computer simulations in injection molding. Imamura et al. (1996) determined PVT relationships at different cooling rates (up to approximately 100 °C/min) and verified their influence on computer simulations. Lobo (1997) attempted to measure the specific volume of semicrystalline polymers at higher cooling rates using a combination of conventional PVT apparatus, differential scanning calorimetry (DSC) measurements and the K-System thermal conductivity apparatus.



$P_K$ : pressure during cooling;  $P$ : pressure of isothermal measurement;  $T_s$ : melt temperature;  $T_v$ : temperature of test;  $T_K$ : temperature of the cooling agent ( $T_s \geq T_v \geq T_K$ ).

Fig. 8. PVT testing device at cooling rates developed by Menges and Thienel (1977).

The National Physical Laboratory (NPL) in the UK (Brown & Hobbs, 1998) has demonstrated a method during rapid cooling (up to 250 °C/min) and under high pressure (up to 250 MPa). Chakravorty (2002) in NPL developed the PVT equipment (Fig. 9) for measuring polymer properties at industrial processing conditions based on the piston-die technique. The equipment could reach a maximum cooling rate of 200 °C/min in



combination with maximum pressure of 160 MPa. Luyé et al. (2001) discussed the specific volume measurement methods for semicrystalline polymers in order to obtain reliable data and analyzed the effect of the cooling rate taking into account the thermal gradient in a cylindrical sample. They discussed the PVT measurement methods for semicrystalline polymers needed in order to obtain reliable data. Particularly, the effect of the cooling rate was analyzed, taking into account the thermal gradient in a cylindrical sample. Specific-volume evolutions from 220°C to 50°C for different cooling rates and different pressures were analyzed, revealing that when the data are corrected to eliminate the thermal gradient effect, the transition zone is much narrower than the experimental one. Moreover, the effect of the pressure and the cooling rate can be more accurately evaluated. It was obtained with an apparatus (PVT100) manufactured by the German supplier SWO Polymertechnik GmbH (Krefeld, Germany). In addition, using the piston device produced a higher imposed cooling rate at the sample periphery than did the immersion system: typically up to 30°C/min in the former case and a few degrees min<sup>-1</sup> in the latter case.

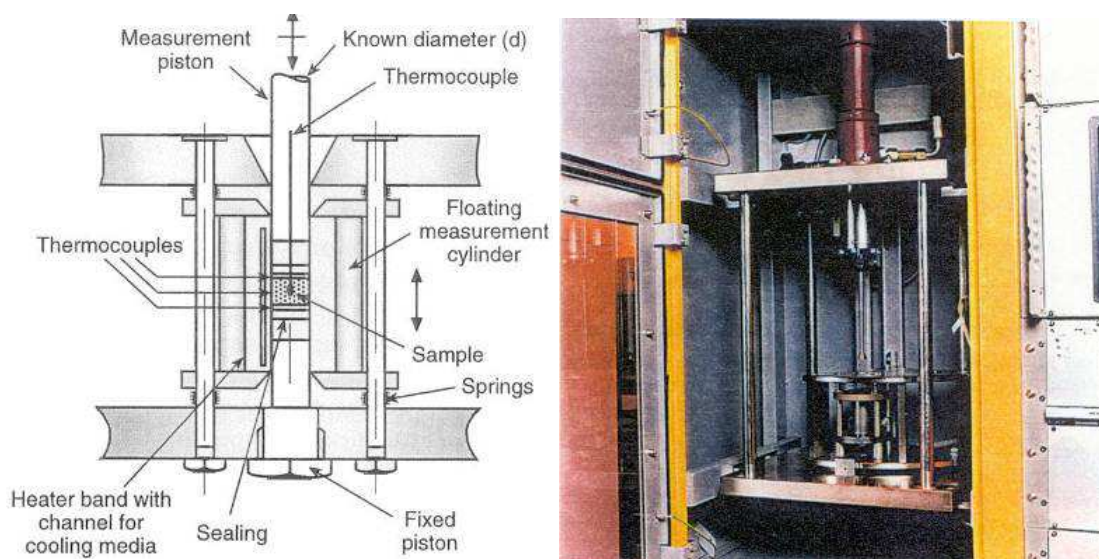


Fig. 9. PVT testing device during rapid cooling and under high pressure developed by NPL (Chakravorty, 2002).

Zuidema et al. (2001) built a setup based on the confining-fluid technique and analyzed the influence of the cooling rate. The setup could reach cooling rates up to 3600 °C/min, but pressure only up to 20 MPa and the accuracy or resolution of the setup was not reported. To minimize the influence of heat on the rest of the measuring equipment, cooling channels were present at the top and the bottom of the heated area to create heat sinks (Fig. 10). When the steady conditions were reached, the vicinity of the sample was quenched with pressurized water via cooling channels positioned close to the sample holding area.

van der Beek et al. (2005a, 2005b) developed a dilatometer to measure specific volume, and the specific volume of polymers as a function of elevated pressure (up to 100 MPa), temperature (up to 260 °C), cooling rate (up to 6000 °C/min) and shear flow in the range of processing conditions as found in injection molding or extrusion. The main goal was not to reach the maximum accuracy possible but rather the ability to analyse specific volume for combination of processing conditions. The dilatometer was based on the piston-die technique in combination with a tensile testing machine with rotation capability. The design

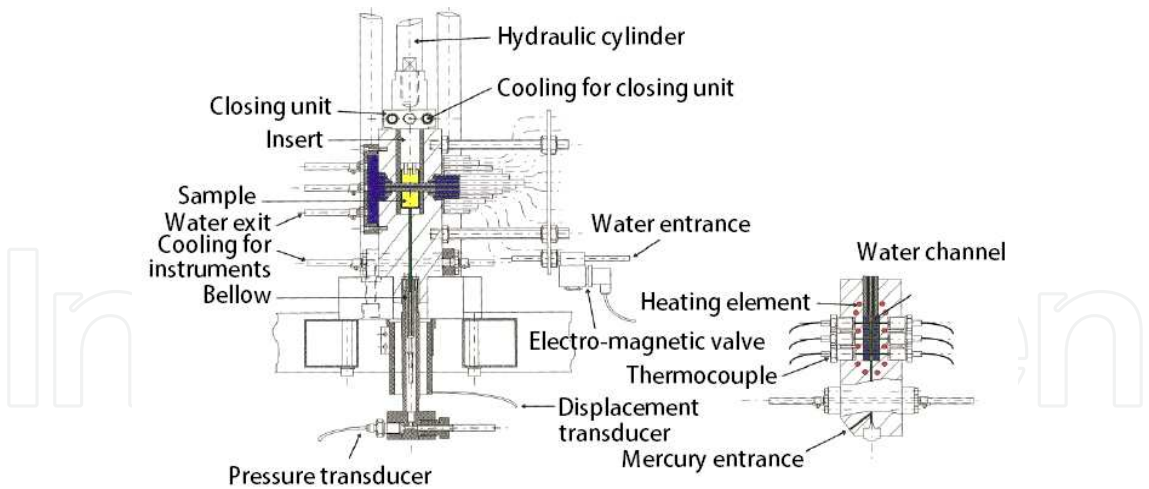


Fig. 10. PVT testing setup developed by Zuidema et al. (2001).

of piston and die was chosen such that an annular shaped sample spacing was created, similar to dilatometers developed by Chakravorty (2002). The particular sampler shape has the advantage that the radial thickness can be chosen small to enable rapid cooling without introducing thermal gradients. Fig. 11 shows a schematic drawing of the pressure cell as mounted to the tensile testing machine.

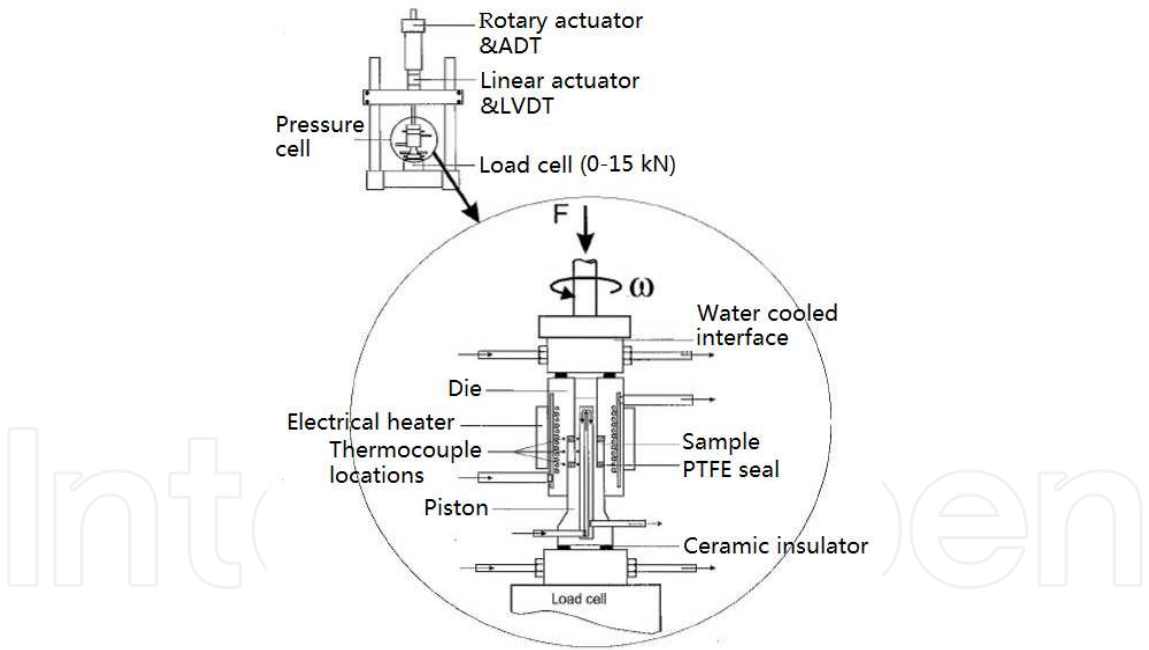


Fig. 11. PVT testing setup developed by van der Beek et al. (2005a).

2.3 On-line techniques

The PVT relationships were almost measured by a special dilatometer. Actually, the technology for measuring PVT relationships using the IMM (injection molding machine) or extruder, which can be called an on-line measurement, is a potentially powerful tool for programming process controllers, because normal process conditions of injection molding or extrusion can be obtained.

Nunn (1989) utilized an IMM to develop PVT properties for a given material by pressurizing the material in the barrel against the blocked outlet nozzle, measuring the volume during pressurization. The injection outlet of the molding machine barrel was blocked and a shot of material was plasticated in the barrel and then pressurized in the barrel at a certain temperature and pressure by axially advancing the screw toward the blocked outlet. The volume of the pressurized shot was then measured by measuring the axial position of the screw. The pressurized shot was then purged and the purged shot was weighed. The specific volume of the material was calculated as the ratio of the shot volume divided by its weight. These steps were then repeated for a plurality of different temperatures and pressures. However, the testing accuracy was not much good.

Chiu et al. (1995) established a method for generating PVT data for thermoplastics on a microcomputer-controlled IMM, which utilized the injection barrel of the IMM as a pressure chamber for determining the specific volume of thermoplastics at various pressure and temperature conditions. The quantities measured with this apparatus were the hydraulic pressure, the barrel temperature, and the screw position. The hydraulic pressure was controlled by adjusting the openings of the relief valve and the servovalve. The specific volume of the polymer in the barrel was calculated from the response of the screw position sensor. A schematic diagram of the control system is given in Fig. 12.

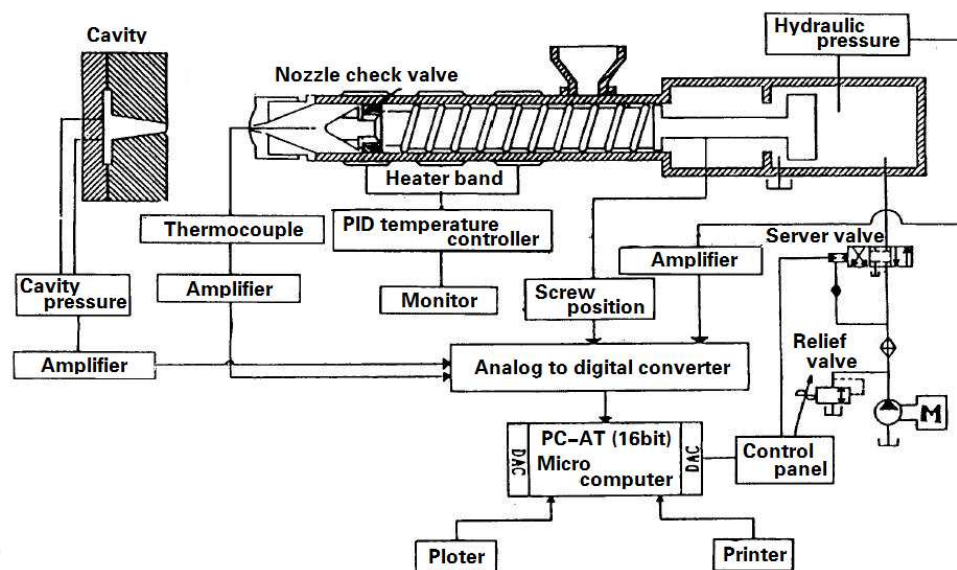


Fig. 12. Microcomputer-controlled injection molding system developed by Chiu et al. (1995).

Park et al. (2004) presented a dilatometer that can measure the PVT properties of polymer melt in a molten state using a foaming extruder and a gear pump. They developed this on-line experimental apparatus, based on an extrusion system. The basic rationale is the determination of the specific volume of the polymer melt by measuring the volume and mass flow rates separately, while controlling the pressure and temperature in the extrusion system independently. Fig. 13 shows a schematic of the designed PVT measurement system. Two extruders were used. The first extruder of the tandem extrusion system plasticated polymer pellets into a melt. The second extruder of a tandem system was used to uniformly lower the melt temperature, while building up the pressure and reducing the fluctuation of pressure. The volumetric flow rate can be determined by measuring the rotational speed of a gear pump due to its positive displacement nature for a polymer melt.



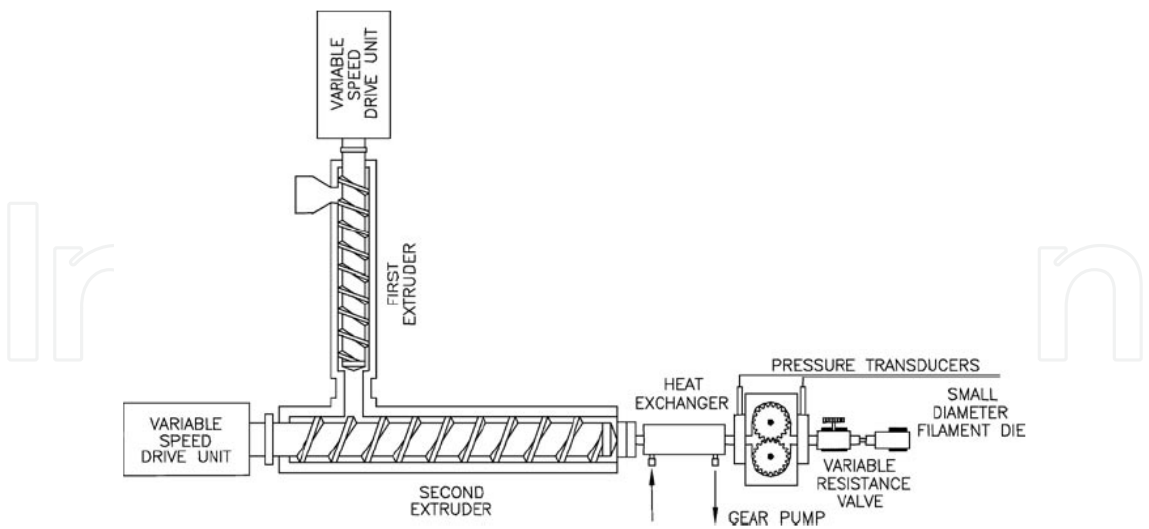


Fig. 13. Testing setup for PVT data of a Polymer/CO<sub>2</sub> solution developed by Park et al. (2004).

All of the experimental results of those above experiments were significantly limited by the machines, especially the maximum pressures were only 9.646 MPa (Nunn, 1989), 96.44 MPa (Chiu et al., 1995) and 28 MPa (Park et al., 2004), respectively. Wang et al. (2009) developed a novel method for testing PVT relationships of polymers based on an IMM. The advantage of this testing approach is that it can be used to obtain PVT data of polymers in mold directly by a special testing mold, however the temperature range was limited below 130 °C, and the pressure range was limited up to 120 MPa.

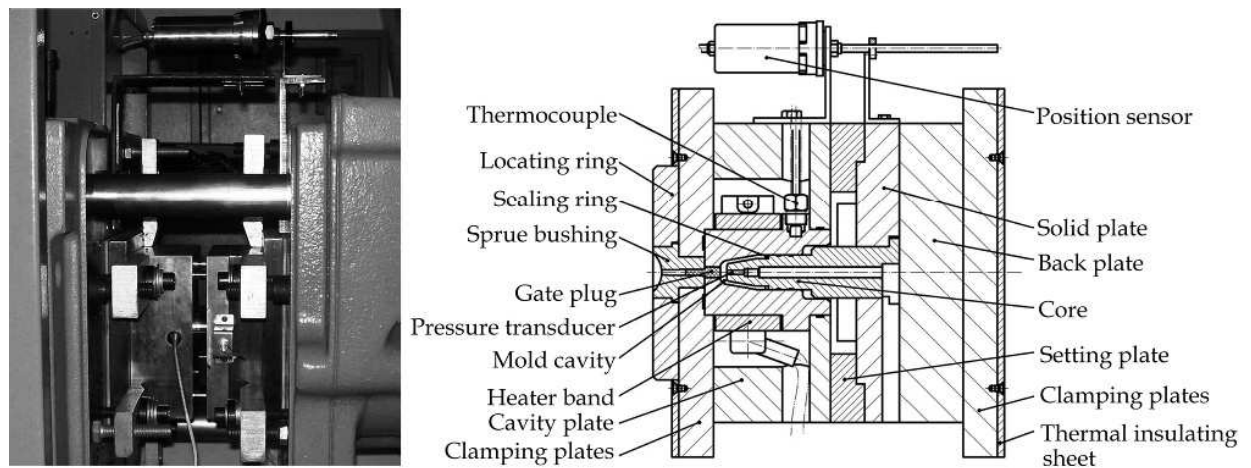


Fig. 14. Picture and detailed outline of the special testing mold (Wang et al., 2010a).

Thus, a new testing mold (Fig. 14) with a small mold cavity was developed to elevate temperature and pressure ranges of the on-line PVT equipment (Wang et al., 2010a), and the heating rate was improved either. This testing mold was assembled onto an IMM to measure the PVT data under normal processing conditions. It used the mold cavity as a pressure chamber for determining the specific volume of polymers under various pressure and temperature conditions. The material was enclosed and pressurized in the rigid mold cavity with a core; the core was close fitting in the mold cavity. During the measuring cycle,

the volume of the material was recorded by the measurement of the displacement of the core. The advantages of this method are:

- The use of the clamping system of the IMM to control the pressure of the sample directly;
- The simplicity in design that can be achieved;
- It is feasible to set parameters directly on the IMM and provide industrial processing conditions;
- The sample is molded and tested directly in an IMM.

## 2.4 Other techniques

There are also some other techniques for measuring the PVT behavior of polymers utilizing ultrasonic technique or X rays. Kim et al. (2004) investigated PVT relationship by ultrasonic technique and its application for the quality prediction of injection molded parts. It proved that the feasibility of ultrasonic response in describing PVT behaviour offered an important basis for monitoring the progress solidification within the mold after injection of polymer melt. Michaeli et al. (2007) developed a new method to determine the specific volume of polymers over a wide range of temperature and pressure based on X-ray attenuation. This method allows the application of different cooling rates enabling the investigation of the density depending on the thermal history.

## 3. Testing modes of polymer PVT properties

There are several testing modes of operation: isothermal compression taken in order of increasing temperature, isothermal compression taken in order of decreasing temperature, isobaric heating and isobaric cooling. Almost all the PVT measurement apparatuses can be used in these several testing modes.

The different testing modes to obtain a PVT diagram can be listed as: (Luyé et al., 2001)

- Isothermal compressing taken in order of increasing temperature (Mode 1): the specific volume is recorded along isotherms (in order of increasing temperature) and at different pressures. Between each temperature there is a stabilization time of a few minutes before the next isotherm. This procedure is often considered the "standard" one.
- Isothermal compressing taken in order of decreasing temperature (Mode 2): the procedure is the same as mode (1), but the isotherms are in order of decreasing temperature.
- Isobaric heating (Mode 3): The specific volume is recorded with a constant heating rate while a constant pressure is maintained and the temperature is varied. When the temperature scan is completed, another pressure is selected and the temperature is varied again.
- Isobaric cooling (Mode 4): The specific volume is recorded along isobars with a fixed cooling rate.

First, since in injection molding the polymer enters the cavity in the melt state and is cooled in the mold, it seems obvious that the transition that must be considered is crystallization. So modes (1) and (2) appear inconvenient because they show the melting transition even if mode (1) is often used.



Second, modes (1) and (2) exhibit a single transition temperature (respectively, melting and crystallization temperatures) independent of the pressure. Nevertheless, this phenomenon is in total contradiction with thermodynamics because both melting and crystallization temperatures are increased by the pressure. Actually, the single observed transition temperature is explained by the following arguments: When the isotherms are followed in the order of increasing temperature, the polymer melts for a given temperature at the lower pressure. Then, when the pressure is increased, it does not have enough time to recrystallize because the crystallization is very slow in this range of temperature. Therefore, the apparent melting temperature on the PVT diagram corresponds to the lowest pressure used in the procedure. When the isotherms are followed in the order of decreasing temperature, due to the thermodynamics, crystallization occurs at a given temperature for the highest pressure (at the end of the isotherm if the pressures are scanned in increasing order). Then, when measuring the next point – that is, the lowest pressure and the next lowest temperature – the polymer is already crystallized, but generally the temperature is lower than the melting point corresponding to this pressure so it cannot melt. Therefore, the apparent single crystallization temperature coincides with the crystallization temperature for the highest pressure used in the procedure. Moreover, since crystallization can be a very slow phenomenon, it can either occur or not occur depending on the chosen stabilization time between two temperatures.

For all the previous reasons, the best procedure seems to be isobaric measurements in cooling mode, mode (4), because in that case the observed transition is the crystallization, and it depends only on the pressure and the cooling rate. The effect of the cooling rate can be investigated. Nevertheless, as it will be shown further, the analysis of the data still faces a difficulty because of the thermal gradient that occurs in the sample, complicating the effect of the cooling rate on the crystallization kinetics.

#### 4. Application of polymer PVT data

In the introduction section, eight major areas were listed for the application of polymer PVT data. Actually, especially for the injection molding, polymer PVT data could be important in two areas: numerical simulation and process control. So in this part, we firstly introduce the equation of state widely used to describe the polymer PVT data, and then show you an example on numerical simulation using different PVT data, at last the development of the control concepts based on the polymer PVT relationship will be introduced.

##### 4.1 PVT equation of state

The equation-of-state (EOS) is very important in describing the thermodynamic properties of liquids and gas-liquid solutions. Spencer and Gilmore (1949) first developed the PVT relations for polymer using a modified van der Waal's equation. The EOS is correlated using the experimental data in a molten state, far above the glass transition temperature. Among the models used to describe the specific volume of polymers in many literatures, the Tait EOS is the most convenient and the most widely one used for polymers. As it was shown by Zoller and Fakhreddine (1994), Tait EOS represents well amorphous as well as semicrystalline polymer melts. It has only been used to describe the PVT behaviour at temperatures above the melting point for polymers. Nonetheless further developed modified 2-domain Tait Equation has been used.

#### 4.1.1 Tait EOS

The PVT relationships can be represented by the 2-domain Tait EOS:

$$V(T,P)=V_0(T)\{1-C \ln[1+P/B(T)]\}+V_1(T,P) \quad (1)$$

where  $V(T,P)$  is the specific volume at temperature  $T$  and pressure  $P$ ,  $V_0$  is the specific volume on the zero gauge pressure,  $C$  is 0.0894 (universal constant), and  $B$  represents the pressure sensitivity of the material. Two temperature domains are required to model the PVT relationship, because the thermodynamic properties of polymers change at the transition to the solid state. The volumetric transition temperature at zero gauge pressure is denoted by  $b_5$ , and the linear increase in the transition with pressure is denoted by  $b_6$ . The specific volume obtained by extrapolating the zero-isobar curve to the transition temperature is denoted by  $b_1$ . This value is the same for both domains when crossing the glass transition. However, when the material is semi-crystalline, the transition due to crystallization is accompanied by an abrupt change in specific volume, such that  $b_{1m}$  (the melt specific volume at  $b_5$  and zero pressure) is greater than  $b_{1s}$ . The temperature dependence of the specific volume is measured by  $b_2$ , while  $b_3$  and  $b_4$  characterize  $B(T)$  in the solid and melt state. The specific volume becomes more pressure sensitive with increasing temperature when  $b_4$  is positive. The constants,  $b_7$ ,  $b_8$  and  $b_9$  characterize  $V_1$  in the solid state.

For  $T < b_5 + b_6P$ :

$$V_0 = b_{1s} + b_{2s}(T - b_5) \quad (2)$$

$$B(T) = b_{3s} \exp[-b_{4s}(T - b_5)] \quad (3)$$

$$V_1 = b_7 \exp[b_8(T - b_5) - b_9P] \quad (4)$$

For  $T > b_5 + b_6P$ :

$$V_0 = b_{1m} + b_{2m}(T - b_5) \quad (5)$$

$$B(T) = b_{3m} \exp[-b_{4m}(T - b_5)] \quad (6)$$

$$V_1 = 0 \quad (7)$$

$b_5$ ,  $b_6$ ,  $b_{1m}$ ,  $b_{2m}$ ,  $b_{3m}$ ,  $b_{4m}$ ,  $b_{1s}$ ,  $b_{2s}$ ,  $b_{3s}$ ,  $b_{4s}$ ,  $b_7$ ,  $b_8$  and  $b_9$  were determined by fitting the experimental PVT data using a nonlinear regression. Software SPSS (SPSS Inc., Chicago, Illinois) could be used for the nonlinear regression. Before the nonlinear regression, the experimental data should be divided into two phases with the transition temperature. With the transition temperature at different pressure,  $b_5$  and  $b_6$  should be calculated firstly; then  $b_{1m}$ ,  $b_{2m}$ ,  $b_{3m}$ ,  $b_{4m}$  in melt state and  $b_{1s}$ ,  $b_{2s}$ ,  $b_{3s}$ ,  $b_{4s}$ ,  $b_7$ ,  $b_8$ ,  $b_9$  in solid state could be calculated separately.

#### 4.1.2 Fitting of PVT data

An example was taken to illustrate the fitting of PVT data. PVT properties of a semi-crystalline polymer, PP (polypropylene), were measured through both the on-line PVT testing mold (Wang et al., 2010a) and a conventional piston-die dilatometer (PVT 100 from SWO Polymertechnik GmbH, 1998). The PVT data of the semicrystalline polymer PP were measured by on-line measurement in the temperature ranges from 17 to 160 °C, pressures from 50 to 200

MPa and heating rate of 15 °C/min; and by piston-die dilatometer in the temperature ranges from 30 to 260 °C, pressures from 20 to 150 MPa and the cooling rate of 2 °C/min. Fig. 15 shows the shape and dimensions of the cup sample used in the on-line measurement. The isothermal compression taken in order of increasing temperatures was applied as the experimental mode of the on-line measurement. The isobaric cooling mode was used by the piston-die dilatometer. The specific volume is recorded along isobars with a fixed cooling rate.

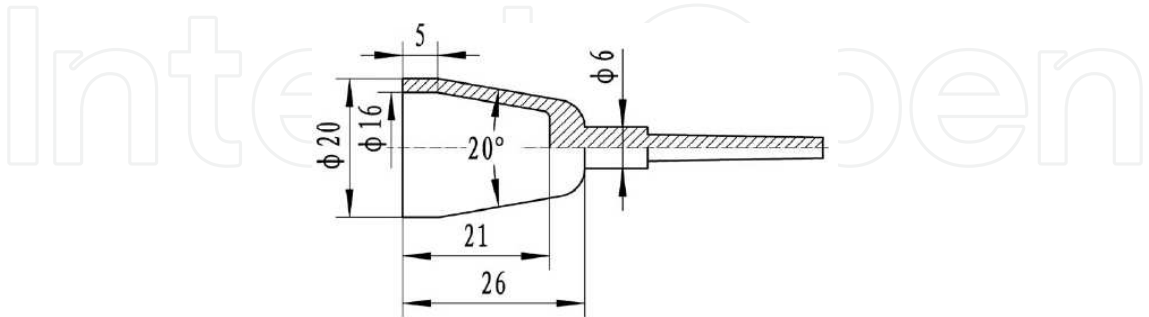


Fig. 15. Shape and dimensions of the cup sample used in the on-line measurement (Wang et al., 2010a).

The experimental results are shown by gray lines and cross dots separately in Fig. 16. The correlation PVT results of both on-line measurement and piston-die dilatometer calculated by Tait EOS are shown by solid lines and dots separately in Fig. 16. The characteristic parameters are listed in Table 1.

Parameter	On-line	Piston-die
$b_5$ (K)	403	417.64
$b_6$ (K/Pa)	0.00000015	3.022E-07
$b_{1m}$ (m <sup>3</sup> /kg)	0.001295	0.001306
$b_{2m}$ (m <sup>3</sup> /(kg · K))	8.588E-07	9.019E-07
$b_{3m}$ (Pa)	77200000	74300000
$b_{4m}$ (1/K)	0.003487	0.004006
$b_{1s}$ (m <sup>3</sup> /kg)	0.001242	0.001195
$b_{2s}$ (m <sup>3</sup> /(kg · K))	9.153E-07	4.929E-07
$b_{3s}$ (Pa)	63200000	131900000
$b_{4s}$ (1/K)	0.006881	0.003355
$b_7$ (m <sup>3</sup> /kg)	0.00002616	0.00009623
$b_8$ (1/K)	0.0714	0.1338
$b_9$ (1/Pa)	3.355E-09	3.973E-08

Table 1. Correlated parameters for the Tait EOS (Wang et al., 2010a).

From Fig. 16, the specific volume of on-line measurement at 20 °C and 0 MPa is 0.64% higher than that of piston-die dilatometer. The specific volume data of on-line measurement increase more with temperature in comparison with those of piston-die dilatometer in a rubbery state, whereas volume change at the melting point temperature of on-line measurement is less than that of piston-die measurement. Above the melting point temperature, although the specific volume of on-line measurement is slightly higher than that of piston-die dilatometer, a difference in the specific volume between them is not significant; the PVT data of the two measurements give a good agreement within 0.045%.

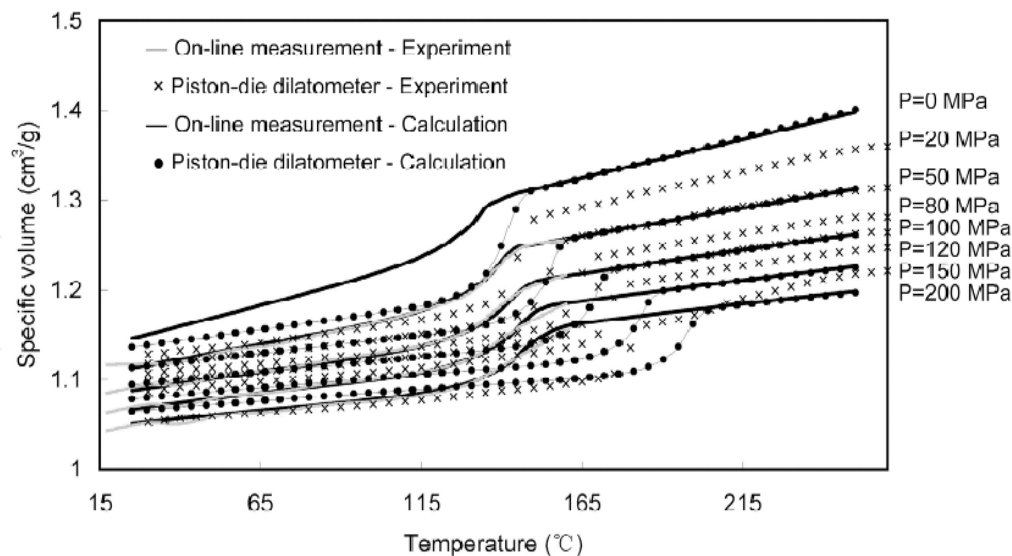


Fig. 16. Experiment and correlation PVT diagrams of PP (Experiment using on-line measurement at  $T=17-160$  °C and  $P=50, 100, 150, 200$  MPa; Experiment using piston-die dilatometer at  $30-260$  °C and  $P=20, 50, 80, 100, 120, 150$  MPa; Correlation using Tait EOS at  $T=20-250$  °C and  $P=0, 50, 100, 150, 200$  MPa) (Wang et al., 2010a).

The reason for the significant difference in the rubbery state must be related to many factors. Because the principles of both of the two measurements are similar, the main differences between them are the different procedures and sample forms. For the procedures, the influence of cooling rate or heating rate is the most important factor. The heating rate of the on-line measurement was  $15$  °C/min; while the cooling rate of the piston-die dilatometer was only  $2$  °C/min. The resulting specific volume in the solid state clearly increases with increasing heating rate.

For the sample forms, the sample of the piston-die dilatometer was a pillar with  $7.4$  mm diameter and  $0.5-1$  g weight, and the sample (Fig. 15) of the on-line measurement was a cup with  $26$  mm length,  $2$  mm thickness and  $2.2-2.4$  g weight. To measure PVT properties accurately, a large amount of samples should be put into the dilatometer. Actually, a sample with large length diameter ratio is the best one to be measured, for it is more convenient and accurate to get the displacement, even using a position sensor with low precision. The thickness of the sample used by on-line measurement was only  $2$  mm. The PVT data thus generated would be closer to typical component part thickness. So the cup sample with  $26$  mm length and  $2$  mm thickness was better for testing than the pillar sample.

#### 4.2 Numerical simulation with PVT data for injection molding

PVT properties of a semicrystalline polymer, PP, were measured through both the on-line testing mold and a conventional piston-die dilatometer. Both of the PVT data were used in numerical simulations for injection molding process. The simulation was carried out by software Moldflow (Autodesk, Inc., San Rafael, CA), and the results of warpage and shrinkage prediction with different PVT data were compared and investigated.

4.2.1 Numerical simulation process with PVT data

The PVT relationships of polymers are the most important factors influencing the shrinkage and warpage of polymer products. In order to show the application of PVT data in numerical simulation and verify the reliability of the PVT results obtained by using the on-line measurement and piston-die dilatometer, prediction of sample shrinkage and warpage was carried out by using Moldflow Plastics Insight (MPI) 6.1. The approach implemented took advantage of the Finite Element (FE) analysis to simulate component fabrication and investigate the main causes of defects. The basic idea is to create a model of the geometry or mold to be analyzed as Fig. 17 shows.

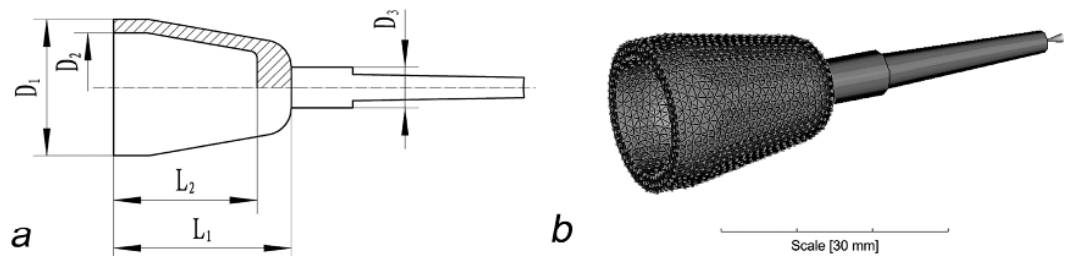


Fig. 17. Principle dimensions (a) and FE mesh (b) of the sample (Wang et al., 2010a).

In order to get an accurate prediction, 3D numerical simulation was used to simulate injection molding process. Fig. 17(a) shows the location of the five characteristic dimensions ( $D_1$ ,  $D_2$ ,  $D_3$ ,  $L_1$  and  $L_2$ ) where results have been analyzed, and Fig. 17(b) shows the FE mesh of the sample. The calculation mesh number is 54338 and the mesh type is four-node tetrahedral element.

Efforts were made to reduce differences in prediction, except the different PVT data, the other input material data such as rheological, thermal and mechanical properties were all measured through the appropriate instruments and kept identical in the simulation. For viscosity, the Cross-WLF model was used, and the flow curves are given in Fig. 18. For PVT data, the modified Tait EOS was used. These other input material data can be referred to Table 2, which were provided by Beijing Yanshan Petrochemical Co., Ltd., SINOPEC.

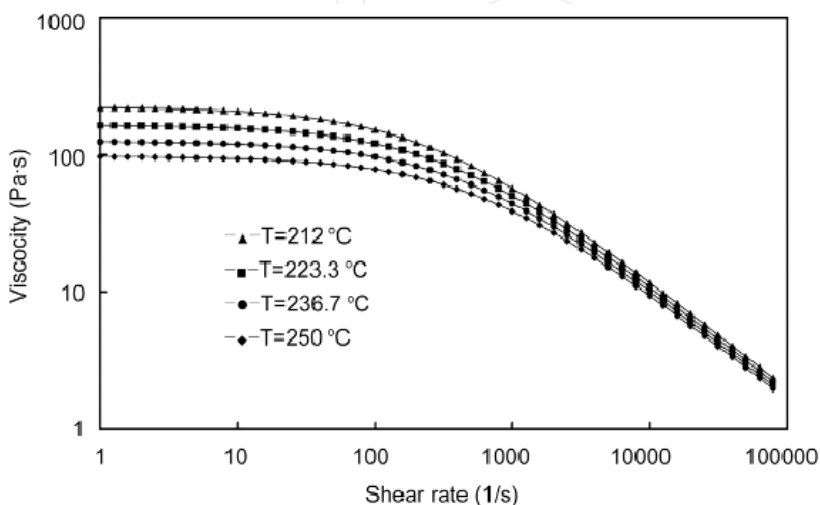


Fig. 18. Flow curves of PP using Cross-WLF model (Wang et al., 2010a).



Mechanical Properties	
Elastic modulus (MPa)	1480
Poissons ratio	0.38
Shear modulus (MPa)	570
Transversely isotropic coefficient of thermal expansion (CTE) data	
Alpha1 (1/°C)	1.07×10 <sup>-4</sup>
Alpha2 (1/°C)	1.07×10 <sup>-4</sup>
Stress at yielde (MPa)	24.8
Strain at break (%)	45
Thermal Properties	
Specific heat data at heating/cooling rate -0.3333 °C/s	
Temperature (°C)	Specific heat (J/kg ·°C)
60	1757
100	2029
150	3158
160	5368
170	4886
200	2259
230	2364
Thermal conductivity data	
Temperature (°C)	250
Thermal conductivity (J/kg ·°C)	0.26
Rheological Properties	
Cross-WLF viscosity model coefficients	
n	0.2065
Tau* (Pa)	57861
D1 (Pa s)	2.25304E+14
D2 (K)	263.15
D3 (K/Pa)	0
A1	34.12
A2 (K)	51.6
Transition temperature (°C)	135
Melt mass-flow rate (MFR)	
Temperature (°C)	230
Load (Kg)	2.16
Measured MFR (g/10min)	45.0

Table 2. Mechanical, thermal and rheological properties of the material (Wang et al., 2010a).  
Similarly, the processing conditions were all identical. As a result, it is possible to focus solely on the PVT data differences. According to the recommended and experimental processing, the process parameters used are reported in Table 3.

Parameter	Value
Volumetric injection rate (cm <sup>3</sup> /s)	30
Mold temperature (°C)	70
Melt temperature (°C)	230
Hold pressure (MPa)	1.8
Hold time (sec)	10
Cooling time (sec)	20

Table 3. Process parameters (Wang et al., 2010a).

4.2.2 Comparison of results with different PVT data

The influence of the PVT data on shrinkage and warpage prediction of the sample was examined. Results for shrinkage and warpage, using Moldflow, are given in Fig. 19 where displacements have been amplified by a factor of 5. The behavior of shrinkage and warpage is observed from the change in dimensions of the sample.

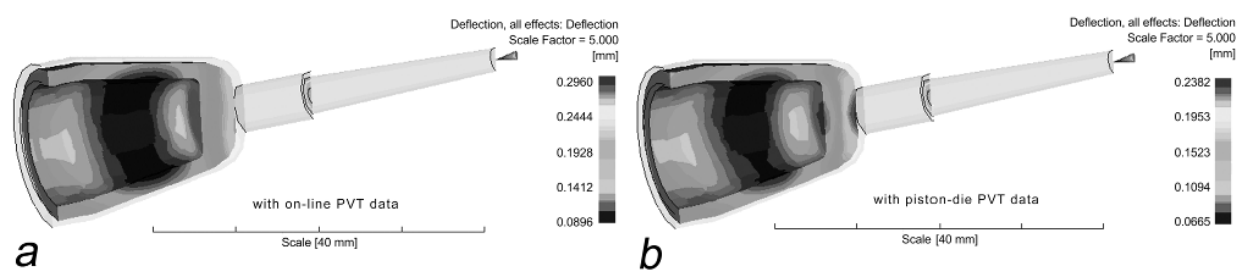


Fig. 19. Numerical simulation of shrinkage and warpage of the sample with on-line PVT data (a) and piston-die PVT data (b) (Wang et al., 2010a).

Table 4 indicates the principle dimensions of the sample measured by micrometer. The experimental shrinkage is 5.3038%, the shrinkage (s) calculation equation is:

$$s = 1 - V_{\text{molding}} / V_{\text{cavity}} \tag{8}$$

In Eq. 8,  $V_{\text{molding}}$  is calculated with the five average dimensions of Table 4,  $V_{\text{cavity}}$  is calculated with the dimensions of the mold cavity.

The experimental results for shrinkage and warpage of final parts are compared with the analysis results of software Moldflow. Shrinkage of the sample is shown in Fig. 20. The quantitative results of shrinkage, 4.557%, calculated by Moldflow with on-line PVT data are observed as being somewhat underestimated in this study, and it is larger than 2.183% analyzed with piston-die PVT data, but much closer to the experimental shrinkage 5.3038%.

Warpage of the sample measured by micrometer and analyzed by Moldflow with both on-line and piston-die PVT data is shown in Fig. 21, and Table 5 indicates the experimental and analyzed deviation value of the principle dimensions. The qualitative description of warpage in this study is sufficiently comparable with the experimental results. The quantitative results are found to be slightly underestimated since the factors which result in warpage have not all been considered. Several factors affect the shrinkage and warpage of molded parts, including molecular orientation, the nonequilibrium state of the polymer PVT behavior, the effect of the flow field on polymer crystallization, anisotropy of material

properties etc., which were considered to accurately describe the real conditions of injection molding as well as to predict the real shrinkage and warpage of molded parts.

	D <sub>1</sub> (mm)	D <sub>2</sub> (mm)	D <sub>3</sub> (mm)	L <sub>1</sub> (mm)	L <sub>2</sub> (mm)
Sample 01	19.60	15.52	5.66	25.42	20.38
Sample 02	19.44	15.50	5.72	25.60	20.50
Sample 03	19.42	15.44	5.68	25.60	20.56
Sample 04	19.48	15.46	5.72	25.50	20.60
Sample 05	19.42	15.44	5.64	25.40	20.50
Sample 06	19.60	15.56	5.66	25.44	20.50
Sample 07	19.42	15.52	5.62	25.62	20.62
Sample 08	19.62	15.58	5.56	25.44	20.34
Sample 09	19.52	15.54	5.70	25.50	20.58
Sample 10	19.40	15.40	5.68	25.60	20.70
Sample 11	19.46	15.50	5.62	25.50	21.00
Sample 12	19.50	15.40	5.62	25.12	20.48
Sample 13	19.48	15.50	5.70	25.50	20.50
Sample 14	19.58	15.54	5.50	25.16	20.74
Sample 15	19.46	15.50	5.78	25.56	20.46
Sample 16	19.46	15.50	5.70	25.52	20.54
Sample 17	19.50	15.50	5.72	25.58	20.48
Sample 18	19.46	15.50	5.64	25.50	20.60
Maximum	19.62	15.58	5.78	25.62	21.00
Minimum	19.40	15.40	5.50	25.12	20.34
Average	19.49	15.49	5.66	25.48	20.56
Cavity	20.00	16.00	6.00	26.00	21.00

Table 4. Principle dimensions of the sample measured by micrometer (Wang et al., 2010).

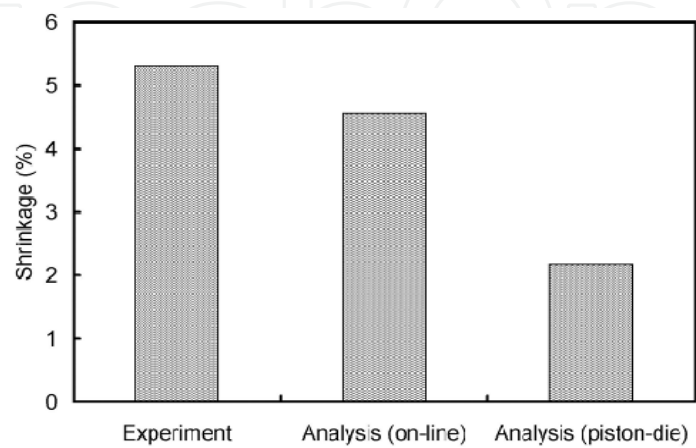


Fig. 20. Shrinkage of the sample (Wang et al., 2010a).

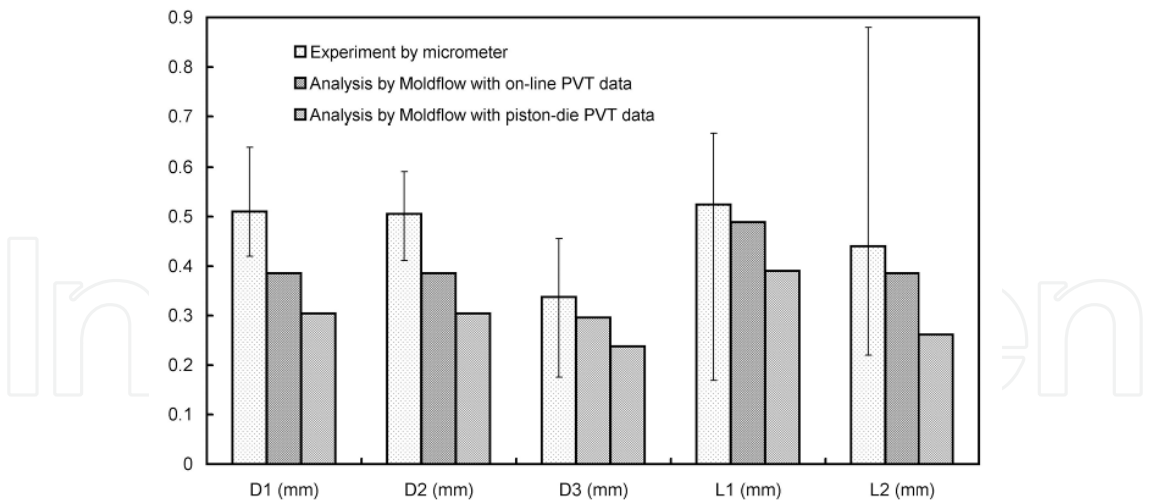


Fig. 21. Comparison of the deviation value of the principle dimensions (Wang et al., 2010a).

	D <sub>1</sub> (mm)	D <sub>2</sub> (mm)	D <sub>3</sub> (mm)	L <sub>1</sub> (mm)	L <sub>2</sub> (mm)
Experiment by micrometer	0.5100	0.505556	0.337778	0.524444	0.4400
Analysis with on-line PVT data	0.3856	0.385600	0.296000	0.488800	0.3856
Analysis with piston-die PVT data	0.3046	0.304600	0.238200	0.390500	0.2617

Table 5. Deviation value of the principle dimensions (Wang et al., 2010a).

For both PVT data, the warpage predictions are very different. Overall, as Fig. 21 shows, the warpage prediction with on-line PVT data is closer to the experimental results than the piston-die testing data.

4.3 Process control with PVT properties for injection molding

The PVT diagram describes the dependence of the specific volume on melt temperature and pressure. It is the basis for a constant quality with the same degree of orientation, residual stresses, and shrinkage which is the goal of isochoric process control (Johannaber, 1994). The application of PVT properties of polymer in process control for injection molding has been studied for years. In this section, injection molding process is described with PVT diagram, and then the development of some process control methods based on the polymer PVT properties are presented.

4.3.1 Description of injection molding process with PVT diagram

Injection molding is a cyclic process consisting of four phases: filling, melt compressing (or packing), holding and cooling, as shown by the typical PVT diagram in Fig.22(a), cavity pressure profile in Fig. 22(b), and cavity temperature profile in Fig.22(c). The filling process starts at Point A. The cavity pressure signal begins at Point B – where the melt plastics touch the pressure sensor for the first time – and then the pressure increases steadily as the filling proceeds. The filling phase is complete at Point C, where the cavity is only volumetrically filled by the melt without being compressed. The packing process then embarks and the pressure rises rapidly to the peak value at Point D. At point D, the injection pressure switches over to the holding pressure and the holding pressure control sets in. Thereafter,

the melt within the cavity is maintained at an assigned pressure during the holding phase, when additional plastic melt can be packed into the cavity to compensate for the plastic shrinkage caused by cooling, so as to have the mold completely filled. This process continues until the gate is frozen, as marked at Point E. Point E is the end-point of the holding phase. Beginning at Point E, a phase of constant volume is maintained (isochoric phase). This isochoric phase is especially important because one strives for a minimum of orientation, residual and distortion. This phase is decisive for the dimensional accuracy of the molding. Reaching Point F in a uniform way is decisive for the constancy of weight and dimensions of the molding. After Point F, the molding cannot be influenced anymore. It shrinks unaffected, usually down to ambient temperature.

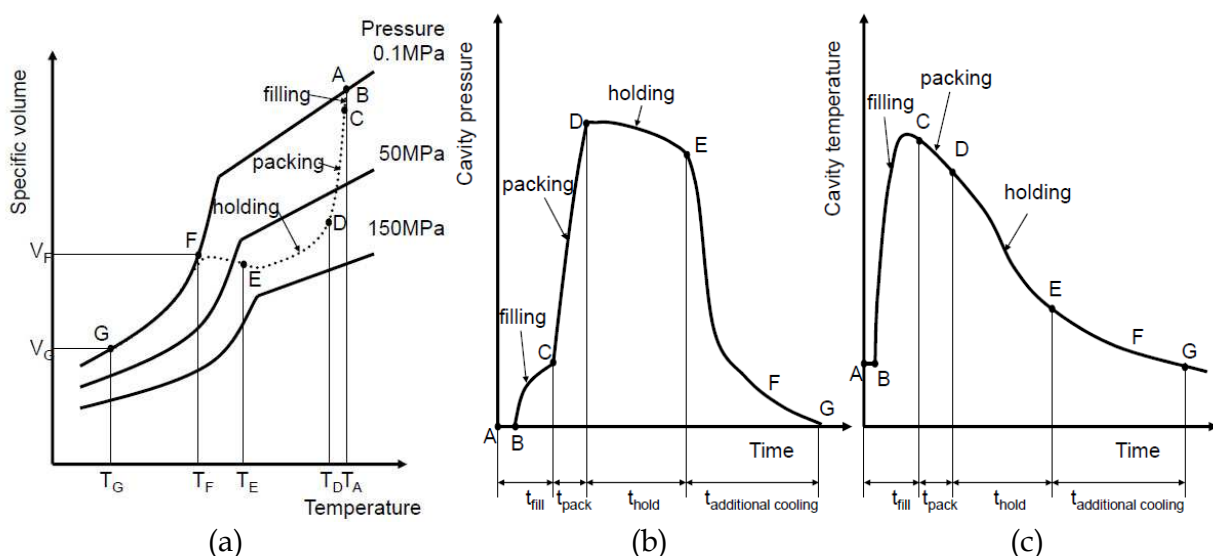


Fig. 22. PVT diagram (a), cavity pressure profile (b) and cavity temperature profile (c).

Therefore, Point D and E are the important transfer points to be controlled in order to obtain optimized holding phase control. There are many methods and theories on the best way to control the transfer of plastic from the injection unit to the mold cavity. Point D is referred to as the filling-to-packing switchover point. The filling-to-packing switchover control during injection molding plays an important role in ensuring the quality of the molded parts (Huang, 2007). There are many filling-to-packing switchover modes that machines are capable of using today, and these different filling-to-packing switchover modes were investigated and compared by some researchers (Edwards, 2003; Chang, 2002; Kazmer et al., 2010). However, it only affects one critical control point in the whole injection molding process. Wang et al. (2011) investigated the filling-to-packing switchover mode based on cavity temperature, and it was proved that the switchover mode based on cavity temperature can be used to accommodate the product weight change due to the variation of mold temperature. Point E is referred to as the holding phase end-point. While the transfer from fill to pack is particularly crucial, transfers from pack to hold and hold to screw recovery also significantly affect part quality. In previous work, authors also studied the end-point control of the holding phase and found that end-point control with cavity temperature can be used to adjust the holding time or cooling time to produce parts with optimum qualities (Wang et al., 2010b). Besides the control of these key transfer points, according to the typical PVT diagram (Fig 22(a)), one way to maintain a high yield rate from



molding is to reproduce the trace from Point A to Point G (especially from Point D to Point E) in every injection molding cycle. Therefore, based on the PVT behaviour of polymer, the qualities of the parts could be optimized.

#### 4.3.2 Development of process control methods with polymer PVT properties

Process variations in the injection molding process can be attributed to a wide variety of possible causes, including process pressure and temperature variations. The cavity pressure profile and its repeatability remarkably influence the quality of the molded part, especially on its weight, dimensional stability, mechanical behavior, and the surface quality (Huang, 2007). Many studies have proposed that the cavity pressure profile can be used to maintain high quality product and help to control the machine in the injection molding process (Huang, 2007). Gao et al. (1996) studied the dynamic behavior and control of cavity pressure for the filling and packing phases. It indicated that reproducing the cavity pressure curve in every shot is one way to maintain the best shot to shot consistency. However, the cavity temperature could not be negligible.

The influence of the cavity temperature on the production rate and the stability of the injection molding process, as well as on the quality of moldings, has been being investigated for a number of years. The cavity temperature influences the following: surface quality, after-shrinkage, orientation, residual stresses and the morphology of polymers (Čatić, 1979). The lower the cavity temperature, the higher the orientation, residual stresses, density of the plastic products, and the lower the surface quality (Menges et al., 1974). Irregularities in cavity temperature profiles from shot to shot can result in defects in the product such as poor mechanical behavior due to residual stresses and other defects causing warpage and differential shrinkage. The weight of the final product and the time required to cool the product are also affected by these cavity temperature profiles (Manero, 1996). However, the cavity temperature changes very fast from melt temperature to the ejected temperature during injection molding, it is difficult to control the cavity temperature exactly in a short time because of the slow response of most thermocouples or temperature sensors.

Therefore, a robust process control technique for achieving high process capabilities which is related to both the cavity pressure and temperature should be developed. It needs to take into account the actual behaviors of the melt plastics being injected into the cavity. The PVT diagram describes the dependence of the specific volume on melt temperature and pressure. It is the basis for a constant quality with the same degree of orientation, residual stresses, and shrinkage which is the goal of isochoric process control (Johannaber, 1994). The application of PVT properties of polymer in process control for injection molding has been studied for years. As the above section, based on the PVT behaviour of polymer, the qualities of the parts could be optimized. The quality of molded parts is primarily determined by the process inside the cavity. The variables of state, such as the pressure, temperature, and specific volume, play an important role (Menges, 1974). Yakemoto et al. (1993) had described an adaptive holding pressure control based on the prediction of polymer temperature in the mold cavity. In their study, they regarded temperature variations as a primary cause of fluctuations in product quality. Their results indicated a strong correlation between the temperature variations and product quality. Sheth and Nunn (1998, 2001) investigated an adaptive process control system to compensate for melt

temperature variations that can occur from shot to shot. The pressure compensation phase of the molding process is modified to compensate for the melt temperature (barrel temperature) change in an effort to achieve dimensional consistency. Kamal et al. (1999) proposed two methods for the control of part weight in injection molding by controlling the peak melt pressure in the cavity and the estimated bulk temperature of the polymer in the cavity at the time when the gate freezes. Both control methods reduced variance in part weight. Michaeli et al. (2004, 2009) developed an online cavity pressure controller, which realized a constant gradient of the cavity pressure during the filling phase and calculated the cavity pressure with a PVT optimization in the holding pressure phase. They indicated that the specific volume displays a significant dependence on the viewed disturbance variables, in particular the melt/mold temperature.

## 5. Summaries

This chapter provided an introduction to different measurements of polymer PVT properties and the application of polymer PVT data for injection molding. Different measurements of polymer PVT properties includes conventional measurements (piston-die technique and confining-fluid technique), some improved experimental techniques considering the effect of cooling rate, shear rate and pressure, on-line techniques using injection molding machine or extruders, etc. Several testing modes of operation were discussed, including: isothermal compression taken in order of increasing temperature, isothermal compression taken in order of decreasing temperature, isobaric heating and isobaric cooling. Almost all the PVT measurement apparatuses can be used in these several testing modes. For injection molding, polymer PVT data could be important in two areas: numerical simulation and process control. So the 2-domain Tait EOS which is used widely in injection molding was introduced in this chapter, then an example on numerical simulation using different PVT data was shown, at last the development of the control concepts based on the polymer PVT relationship was introduced.

From these different research fields, we can see that the PVT properties of polymers play the most important role in both numerical simulation and process control for injection molding. The knowledge on PVT properties of polymers could be the fundamental concepts for the engineers in injection molding.

## 6. Acknowledgments

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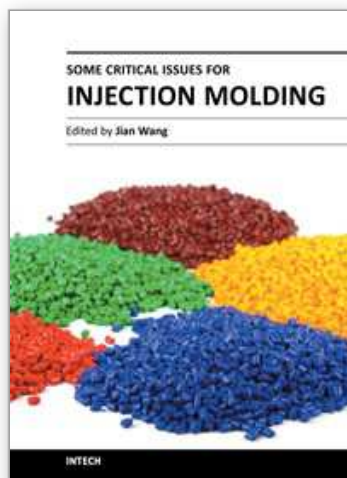
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## **Some Critical Issues for Injection Molding**

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This book is composed of different chapters which are related to the subject of injection molding and written by leading international academic experts in the field. It contains introduction on polymer PVT measurements and two main application areas of polymer PVT data in injection molding, optimization for injection molding process, Powder Injection Molding which comprises Ceramic Injection Molding and Metal Injection Molding, and some special techniques or applications in injection molding. It provides some clear presentation of injection molding process and equipment to direct people in plastics manufacturing to solve problems and avoid costly errors. With useful, fundamental information for knowing and optimizing the injection molding operation, the readers could gain some working knowledge of the injection molding.

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Unit 405, Office Block, Hotel Equatorial Shanghai  
No.65, Yan An Road (West), Shanghai, 200040, China  
中国上海市延安西路65号上海国际贵都大饭店办公楼405单元  
Phone: +86-21-62489820  
Fax: +86-21-62489821

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